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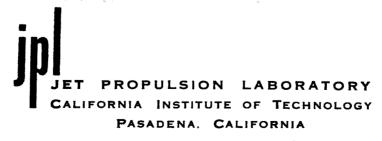*Technical Report No. 32-359*

Tensile Properties of Tungsten from 2500 to 5400 F in Vacuum

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ABSTRACT.   The effects of temperature and strain rate on the tensile properties of recrystallized powder metallurgy tungsten rod were studied.   Tests were conducted on a standard specimen (length/diameter $\simeq$ 4) at strain rates of 0.02 and 2.0 per min.   The strain-hardening exponent was determined to vary between 0.075 and 0.90, increasing with increasing strain rate and decreasing temperature. Intercrystalline-type fracture was observed at the lower strain rate at all temperatures studied.   This type of fracture is associated with void formation and growth and relatively low ductility values, between 22 and 55%, as measured by reduction in area.   A ductility minimum occurs between 3000 and 4000 F (1650 and 2200 C). Ductile transgranular fracture, with reduction-in-area values between 90 and 100%, was found between 2500 and 5400 F (1370 and 2980 C) at 2.0 per min, an order of magnitude lower strain rate than previously reported by others.   Above 3000 F (1650 C) at the 2.0 per min strain rate, stress-induced grain growth accompanied by a kind of work softening as shown by true stress-strain curves appears to account for the high ductility measured.

Arc-melted and extruded tungsten and plasma-flame single-crystal tungsten in rod form were studied at the lower strain rate.   These materials were found to have lower strength and higher ductility than powder metallurgy tungsten in the temperature range studied.   *ASM-SLA Classification: Q27, Q23p, Q26, 2-61, 3-68; W, 4-55.*

TUNGSTEN, the highest melting metal (6170 F or 3410 C), has received comparatively little research attention at temperatures above 3000 F (1650 C). Low impurity levels are known to markedly affect the ductility (at low and intermediate

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temperatures) of tungsten and its sister metals molybdenum and chromium in Group VIa. Questions about the effect of impurity levels at high temperatures have not been fully answered. Further, the effect of grain boundaries on high-temperature strength is not well understood.

A recent compilation (1) of tensile properties of tungsten shows that considerable scatter exists in strength and ductility data, particularly above 3000 F (1650 C). Arc-melted tungsten in the recrystallized state remains ductile above 3000 F (1650 C), and the powder metallurgy material, as recrystallized, exhibits relatively brittle behavior, particularly at strain rates on the order of 2.0 per min and below. Strain rate has been found (2) to have a large

TABLE 1. Chemical Analysis of Tungsten Materials

Material	Composition, parts per million by weight							
	O	N	C	Fe	Ni	Al	Si	Mo
Powder metallurgy	1.3 – 5	5 –16	2 – 4	10 – 40	<1	20– 40	<10– 20	30 – 50
Arc-melted	1.9 – 32	5 – 9	<2 – 7	4 – 22	*	*	*	*
Arc-melted and extruded	1.2 – 8.5	4 – 8	<2 – 5	<2 – 9	*	*	*	*
Plasma-flame single-crystal	4.9 – 8.7	3 – 7	66 – 98	20 – 40	<10	10 – 30	10 – 30	*

*Not determined.

TABLE 2. Powder Metallurgy Tungsten Tensile Properties at 400 F (200 C)

Condition	Ultimate tensile strength, psi	Reduction in area, %	Elongation, %	Strain rate
As received	119,500	58.2	32.8	0.02/min
Recrystallized*	69,600	6.7	6.2	0.02/min
Recrystallized*	67,200	6.8	6.2	0.02/min

* Recrystallized at 5150 F (2840 C) for 10 min.

effect on strength and ductility; increasing strain rate increases both strength and ductility.

From the scatter within given sets of data and the differences among investigations (2–6), it appears probable that composition, processing history, and testing technique have a significant influence on tensile properties, especially ductility. Testing techniques employed by others often involve self-resistance, radiation, or electron-beam heating of a specimen made especially long to accommodate cold grips. Such techniques are likely to introduce temperature gradients in the gage length of the specimen. Longer-than-standard (≃4 to 1) gage length to diameter ratios make elongation comparisons difficult.

The present investigation was undertaken to establish reproducible tensile data, including stress-strain curves, and to determine the fracture characteristics of commercially pure tungsten. Using a standard specimen in a hot-grip assembly, measurements were made to 5400 F (2980 C). The effect of temperature and strain rate on recrystallized powder metallurgy tungsten was studied. For comparison, to determine possible structure and impurity effects, arc-melted and extruded tungsten and plasma-flame single-crystal tungsten were investigated.

MATERIALS

Results of chemical analyses of the tungsten materials used in the investigation are summarized in Table 1. The powder metallurgy (PM) tungsten was obtained in the form of $\frac{3}{8}$-in. diam swaged rod. The table indicates the range of composition for the four PM rods tested. The material is further characterized in Table 2 by some 400 F (200 C) tensile properties, in the condition noted. By electropolishing, 0.008 in. was removed from the diameter of these specimens prior to testing to minimize surface effects.

Arc-melted and extruded (AM & E) material was made by arc-melting $\frac{3}{4}$-in. diam swaged rod. The $1\frac{3}{8}$-in. diam castings were extruded to $\frac{3}{8}$-in. diam rod in a high-energy-rate extrusion machine. Analyses (Table 1) for the arc casting and the subsequent extrusion (after surface cleanup) are given to show that the composition was essentially unchanged.

Two plasma-flame (PF) single-crystal rods, nominally $\frac{7}{8}$ in. in diameter by 12-in. long, were used in this study. The tensile axis was determined to be parallel within two degrees of the <111> direction in both crystals. With the exception of carbon content (Table 1), the composition of the PF crystals did not differ markedly
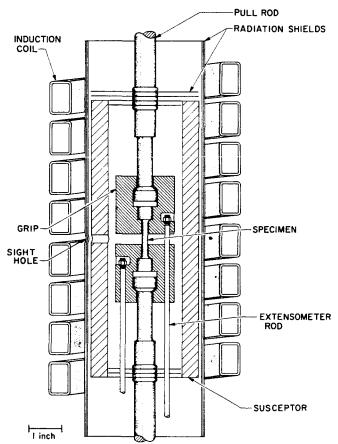
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FIG. 1.   Specimen-grip assembly in susceptor and induction coil.

from that of the other two tungsten materials.

All specimens were heated at 5150 F (2840 C) for 10 min in a vacuum on the order of 10^{-4} torr and then slowly cooled to room temperature prior to testing. This treatment resulted in an average grain size as determined by the intercept method of 240 grains per mm² for the PM specimens and 10 grains per mm² for the AM & E tungsten.

Apparatus and Procedures

The tensile apparatus and procedures developed for this investigation have been described in detail (7). Principal features of the apparatus include a hot-grip assembly heated by radiation from an induction-heated susceptor, shown in Fig. 1; a dc gear motor-driven lead screw giving strain rates from 0.005 to 5.0 per min on a 0.640-in. gage length specimen; and load-elongation recording instrumentation. Vacuum pumping on a water-cooled chamber enclosing the heater assembly provides pressures between 2×10^{-4} and 9×10^{-4} torr. Vacuum is measured by a hot-filament ion gage located in a 4-in. pipe elbow between the chamber and the diffusion pump.

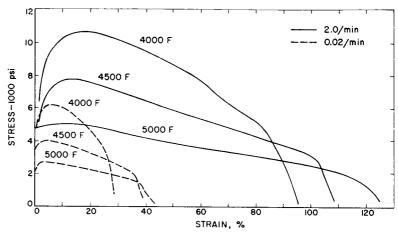Temperature measurements are made

FIG. 2. Typical engineering stress-strain curves for recrystallized powder metallurgy tungsten at two strain rates.

with a disappearing filament-type optical pyrometer. The hot-zone enclosure has been shown to be sufficiently near black-body conditions (7) to omit an emissivity correction. Over-all accuracy in temperature measurement is considered to be ±30 F (±18 C). No temperature gradient exists in the specimen within the limit of detection of the pyrometer, that is 10 F (6 C).

The specimen conforms to the R-4 type (8) with a diameter of 0.160 in. and a length-to-diameter ratio of approximately four. The seat in the grip is a negative replica of the specimen shoulder fillet.

The heating rate is dependent on the capacity of the vacuum system to maintain the desired pressure while handling the out-gassing load due to heating. As a result, $3\frac{1}{2}$ hr are required to reach 5400 F (2980 C). The specimen is held 10 min at test temperature, then strained at constant lead-screw speed to fracture.

RESULTS AND DISCUSSION

Stress-Strain Curves and Strain-Hardening Exponent

Typical engineering stress-strain curves for recrystallized PM tungsten tested at two strain rates, 0.02 and 2.0 per min, at 4000, 4500, and 5000 F (2200, 2480, and 2760 C), are presented in Fig. 2. These curves, as compared to room-temperature stress-strain curves for most materials, present some unusual features. At the 2.0 per min strain rate the onset of necking is not associated with the maximum load, but with much lower loads ($\simeq$50%) at strains on the order of 100%. The curves at the 0.02 per min strain rate, although similar in shape, show considerably less strain. Fracture occurs without necking.

The features of the engineering stress-strain curves suggest looking at true-stress –true-strain plots for further insight. Four true-stress – true-strain curves were plotted (Fig. 3) for the 2.0 per min strain rate, assuming constant volume and uniform elongation to the point of necking, which is very nearly true. Normal behavior is exhibited at 2500 F (1370 C) and seems to be associated with a lack of stress-induced grain growth. As discussed later, grain growth does not occur below 3000 F (1650 C) at this strain rate. The curves at higher temperatures have slightly negative to nearly level slopes in the strain region after maximum strain. This implies
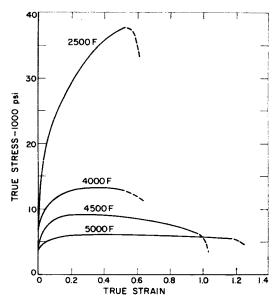
FIG. 3. True stress as a function of true strain at a strain rate of 2.0/min and various temperatures for recrystallized powder metallurgy tungsten.

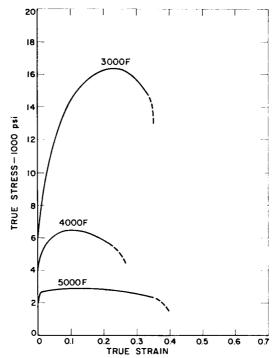some form of work softening possibly associated with grain growth.

The true-stress – true-strain curves for the 0.02 per min rate are given in Fig. 4. Constant volume is not maintained during uniform elongation because of internal void formation ranging from 3 to 8 vol %. A correction for void volume was made when plotting the curves. The shape of the curves is consistent with that at the higher strain rate (Fig. 3) in that normal behavior is seen at 3000 F (1650 C), and transition behavior to work softening at 4000 F (2200 C) and above, where grain growth takes place.

One important parameter obtained from a true-stress-true-strain curve is the strain-hardening exponent. A true-stress – true-strain curve or flow curve may be expressed by the equation $\sigma = K\epsilon^n$, where σ is the true stress, K is the strength coefficient, ϵ is the true strain, and n is the strain-hardening exponent. Thus n is the slope of the nearly straight line obtained from a log-log plot of true stress and true

strain up to the point of maximum stress. Values of n are plotted as a function of temperature for two strain rates in Fig. 5. The value of n decreases with increasing temperature and decreasing strain rate. Two interesting points are noted in these data. First, although n is still decreasing at 5400 F (2980 C) for the high strain rate, n appears to reach a minimum value of 0.075 at 4500 F (2480 C) for the low strain rate. Second, a departure from a smooth curve occurs at approximately 3500 F (1930 C) for both strain rates. This deviation may have significance, for it is in this temperature region that the ductility minimum is found at the 0.02 per min-strain rate and grain growth occurs at the 2.0 per min-strain rate. To the authors' knowledge no data are available for comparison.

Tensile Properties of PM Tungsten

Tensile data for the PM tungsten are presented in Fig. 6, 7, and 8. Results of

FIG. 4.   True stress as a function of true strain at a strain rate of 0.02/min and various temperatures for recrystallized powder metallurgy tungsten.
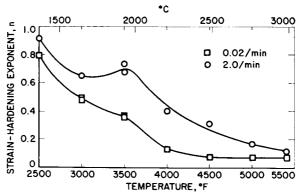

FIG. 5.   Strain-hardening exponent, n, as a function of temperature at two strain rates for recrystallized powder metallurgy tungsten.

Sikora and Hall (2) for $\frac{1}{8}$-in. swaged PM tungsten rod, recrystallized for 1 hr at 4050 F (2230 C), are included for comparison. The narrow scatter band of the present data points indicates excellent data reproducibility, considering the experimental difficulties in this temperature region.

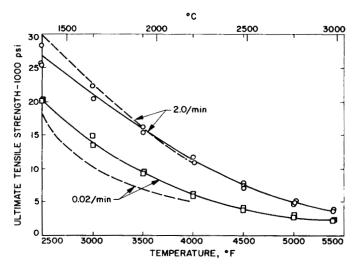As shown by Fig. 6, the ultimate tensile

°C

FIG. 6.   Ultimate tensile strength as a function of temperature for recrystallized powder metallurgy tungsten at two strain rates.   Dashed lines are data from Ref. 2.
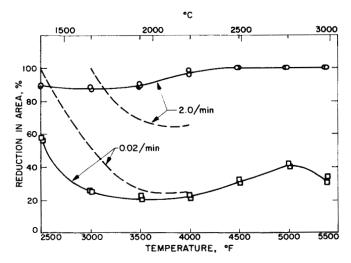
°C

FIG. 7.   Reduction in areas as a function of temperature for recrystallized powder metallurgy tungsten at two strain rates.   Dashed lines are data from Ref. 2.

strength is strain-rate dependent. The present data agree with previous work (2) on PM tungsten at 2.0 per min, but differ somewhat at the lower strain rate.

Ductility is found to have a greater strain-rate dependence than strength. Reduction-in-area values (Fig. 7) in the present work show a notable deviation from the comparison data at the higher strain rate. Values of 90% or greater
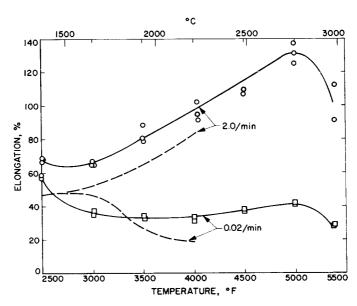
FIG. 8. Elongation as a function of temperature for recrystallized powder metallurgy tungsten at two strain rates. Dashed lines are data from Ref. 2.
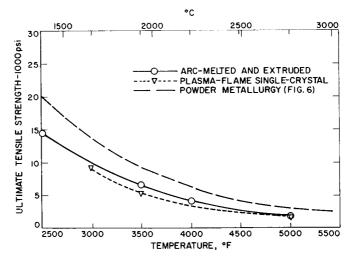

FIG. 9. Ultimate tensile strength as a function of temperature for arc-melted and extruded tungsten and plasma-flame single-crystal tungsten at the 0.02/min strain rate.

were found over the entire temperature range. At the 0.02 per min strain rate the curves differ, principally below 3500 F (1930 C), but both show a ductility mini- mum between 3000 and 4000 F (1650 and 2200 C). Elongation data (Fig. 8) also differ in magnitude although the general trend is similar. Elongation reaches a
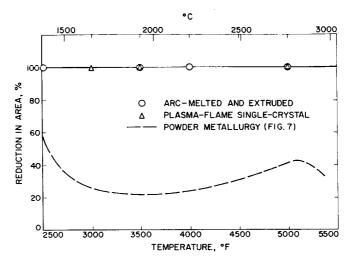
°C

FIG. 10. Reduction in area as a function of temperature for arc-melted and extruded tungsten and plasma-flame single-crystal tungsten at the 0.02/min strain rate.

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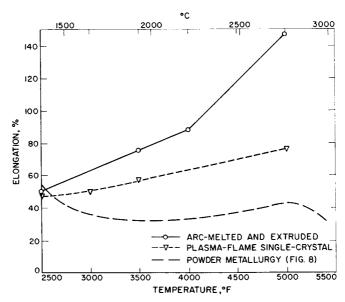
FIG. 11. Elongation as a function of temperature for arc-melted and extruded tungsten and plasma-flame single-crystal tungsten at the 0.02/min strain rate.

for formation of voids. The actual temperature at which grain-boundary sliding occurs is strain-rate-dependent, decreasing with decreasing strain rate, and is also structure-sensitive.

The upper temperature limit of the

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maximum of 130% at 5000 F (2760 C) for the 2.0 per min strain rate and then decreases. Only Glasier (5) has reported a drop in elongation values between 4000 F (2200 C) and 6000 F (3310 C). At the 0.02 per min strain rate, reduction in area and elongation increase between 3000 and 5000 F (1650 and 2760 C) and decrease somewhat at 5400 F (2980 C). A decrease in reduction in area at high temperatures has not previously been reported.

Ductile transgranular fracture was observed at the 2.0 per min strain rate and is consistent with the high elongation and reduction-in-area values obtained. Intercrystalline fractures noted at the 0.02 per min strain rate are accompanied by void formation at grain boundaries perpendicular to the tensile axis. Voids, not present after recrystallization, are nucleated and grow throughout the gage length during testing. Fracture occurs at low values of engineering stress because of a reduced cross-sectional area and possible internal stress concentration. Little or no necking accompanies fracture.

Tensile Properties of PF and AM & E Tungsten

Data for AM & E and PF single-crystal specimens at the 0.02 per min strain rate are given in Fig. 9, 10, and 11. Both materials show lower strength and higher ductility than PM tungsten, with a complete absence of intercrystalline fracture at all temperatures, as indicated by the 99+% reduction-in-area values obtained. The AM & E tungsten, as compared to the PF single-crystal tungsten, exhibits a higher elongation (Fig. 11) and slightly higher strength (Fig. 9), presumed to be related to composition, orientation, and grain-boundary effects. It is only in the elongation curves (Fig. 11) that a difference in ductility is observed between AM & E and PF tungsten.

Effect of Grain Boundaries

The effect of grain boundaries on the high-temperature strength of metals is not completely understood. Possible strengthening effects are shown by the present data. Figure 9 shows that PM tungsten with 240 grains per mm² is slightly stronger than AM & E tungsten with 10 grains per mm². Assuming that grain growth during testing has little effect on ultimate strength (attained early in the deformation cycle) and noting similar material composition (Table 1), it appears that grain boundaries in tungsten have a strengthening effect.

Although the higher carbon content of PF tungsten might be expected to exert a small strengthening effect, PF tungsten is slightly weaker than the AM & E material. It is not clear, however, to what extent crystal orientation ($<$111$>$) or the lack of grain boundaries influence the results.

Voids and Intercrystalline Fracture

The absence of voids and of associated intercrystalline fracture at the 2.0 per min strain rate in PM tungsten deserves further consideration because of the profound effect this phenomenon has on mechanical properties, particularly ductility. Although it is known that increasing strain rate decreases the tendency toward void nucleation and growth and intercrystalline fracture, previous data have not shown transgranular fractures in rod specimens at strain rates as low as 2.0 per min.

The ductility of PM tungsten at elevated temperatures has often been said to be low because sintered materials are inherently weak at grain boundaries. It now appears that this low ductility, which is characterized by a minimum, is a manifestation of so-called "intermediate temperature" brittleness. This ductility minimum is associated with intercrystalline-type fracture and observed in many metals at strain rates of 0.02 per min and below.

Present fracture theories (9) describe a critical temperature region for intercrystalline fracture as between approximately $0.5\ T_m$ (temperature of melting, K) and some higher temperature. Grain-boundary shearing or sliding begins at approximately $0.5\ T_m$ and is a prerequisite
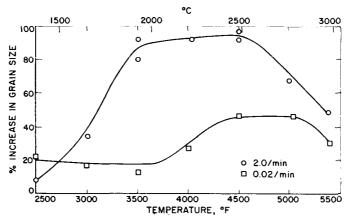
Fig. 12. Percent increase in grain size change during testing as a function of temperature at two strain rates for recrystallized powder metallurgy tungsten.

for grain growth than do PM materials. In the present work, AM & E tungsten shows a knife-edge fracture at 2500 F (1370 C), which is a low temperature for stress-induced grain growth. As chemical analyses show the PM and AM & E specimens to be similar, either exceedingly small impurity levels or their distribution must account for the large differences observed. Having no grain boundaries, PF tungsten would not be expected to show void formation and intercrystalline fracture at any strain rate, and none was observed in the present work.

CONCLUSIONS

1. The tensile properties of several tungsten materials were measured in the temperature range from 2500 to 5400 F (1370 to 2980 C). Individual data points were found to be quite reproducible.

2. A ductility minimum for PM tungsten was found to lie between 3000 and 4000 F (1650 and 2200 C) at the 0.02 per min strain rate, confirming the work of previous investigators. This minimum is considered from present work to be the same "intermediate temperature" brittleness observed above 0.5 T_m in many metals. Grain-boundary sliding in this temperature region is thought to nucleate voids which grow by

a stress-induced vacancy redistribution process.

3. Experimental data show that ductility is regained in PM tungsten when the temperature and strain rate are such as to produce stress-induced grain growth. It is suggested that grain growth during testing controls or limits void growth. Stress-induced grain growth is strain-rate-, temperature-, and impurity-level-dependent.

4. The negative slope of the true-stress – true-strain curves for PM tungsten in the uniform elongation region following the maximum strength indicates that some form of work softening is occurring, probably associated with grain growth.

5. The significant differences in ductility between arc-melted tungsten and powder metallurgy tungsten can be explained by the stress-induced grain growth process which occurs more readily in melted material.

6. It appears that grain boundaries contribute to strength in tungsten in the temperature range studied.

ACKNOWLEDGMENT

ductility-minimum region has been associated with recrystallization or grain growth. At elevated temperatures, the ease of boundary movement is thought to prevent the necessary stress concentration for void nucleation and growth; either the boundaries move before sufficient stress can be built up to nucleate a void or the boundary moves some time during the void growth cycle, thus preventing further growth.

Stress-Induced Grain Growth

With grain-boundary shearing and movement and their possible effects in mind, a study of the microstructures of the fractured specimens was made. This study revealed the occurrence of stress-induced grain-boundary motion or grain growth above 3000 F (1650 C) at the 2.0 per min strain rate and above approximately 4000 F (2200 C) at the slower rate of testing. Figure 12 summarizes these data, presenting the increase in grain size during testing as a function of test temperature for the two strain rates. Percent increase in grain size is defined as the difference in grain size between the specimen head and the uniformly deformed gage section away from the fracture, divided by grain size in the head, multiplied by 100. The effect of temperature alone is excluded by referencing the measurements to the grain size in the undeformed head.

Stress-induced grain growth is seen to be a strong function of strain rate and temperature. Above approximately 4500 F (2480 C) grain growth is observed to diminish at both strain rates. Although the causes of this decrease are unknown, extremely high recovery rates at these temperatures may limit the storing of energy available for motion.

The ductile fracture shapes correlate well with the grain growth observed. A shear fracture at 2500 F (1370 C), where little grain growth is observed, changes to a knife-edge type more characteristic of single crystals above approximately 3500 F (1930 C). Little difference in the shape

of the fractures of the low-ductility specimens was noted even above 4500 F (2480 C).

A similarity is noted in three curves at the lower strain rate: grain-size change (Fig. 12), reduction-in-area (Fig. 7), and elongation (Fig. 8), all plotted as a function of temperature. In the temperature region where growth is occurring, ductility is increasing, as shown by the reduction-in-area and elongation curves.

If the kinetics of stress-induced grain growth were known, it might be possible to say which of the two processes, void growth or grain growth, is controlling ductility. In other words, is void growth limited by the onset of grain growth, or grain growth limited by the presence of voids and possibly the total strain? A study directed to this question has only recently been undertaken at this laboratory.

The experimental evidence suggests that grain growth, under the conditions where it occurs, limits void growth and therefore intercrystalline fracture. At the high strain rate very few voids are found, while at the low strain rate grain growth occurs in the presence of voids at and above 4500 F (2480 C) as shown in Fig. 12. The higher stresses required for plastic flow at the higher strain rate can account for the occurrence of boundary motion at lower temperatures. Variations in impurity level or distribution may result in a different strain-rate dependence of stress-induced grain growth for various tungsten materials.

Below 3000 F (1650 C) high ductility at both strain rates is observed without grain growth during testing. The lack of grain-boundary sliding and hence void formation at these temperatures (below 0.5 T_m) is thought to account for the high ductility found.

The absence of void growth and intercrystalline fracture in AM & E tungsten at 0.02 per min strain rate is consistent with the idea of void growth being limited by boundary motion. Arc-melted materials are known to exhibit a greater tendency

Martens for many helpful suggestions and to Messrs. Harry Tracy and Warren Torris, who were responsible for the experimental data.

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